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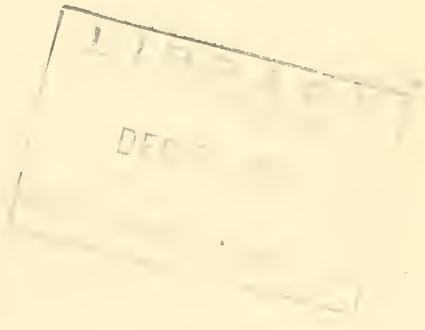
A PROPOSED STANDARD FOR DESIGNATION OF "CLOUD" IN CITRUS JUICES



Agricultural Research Service
UNITED STATES DEPARTMENT OF AGRICULTURE

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A PROPOSED STANDARD FOR DESIGNATION OF "CLOUD" IN CITRUS JUICES

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INTRODUCTION

The development of any product inevitably poses new problems of quality control, and frozen citrus juice concentrates are not exceptions. Control of quality at the ultimate consumer level, rather than quality at the point of production only, should be of prime importance to the producer of any food product. While a product of the highest quality may be produced by a manufacturer of frozen citrus juice concentrate, changes during subsequent distribution through normal channels or during storage in the housewife's kitchen may result in an unacceptable product on the table. Among the changes which take place during improper handling or storage of frozen citrus juice concentrates are clarification, gelation, and undesirable flavor changes. At least some of these changes are enzymatic in nature and can be expected to occur if the product is allowed to remain for unduly long periods at temperatures more than a few degrees above zero degrees Fahrenheit.

One of the important characteristics of a citrus juice or of reconstituted citrus concentrate is the turbidity or "cloud" normal to these juices. It is fortunate that this cloud can be measured readily, since it is commonly thought in the industry that partial loss of cloud is the first noticeable sign of degradative change in juice products (1)[2]. This cloud probably consists of the colloidally dispersed cell fragments remaining after the coarser cell debris is removed by centrifugation. In order to insure reproducible measurement of the cloud, the juice or reconstituted concentrate is centrifuged under defined conditions. The residual turbidity is commonly determined with a photoelectric colorimeter. A red filter is used to eliminate interference from the normal pigments of the juice, since these do not absorb light in the red region of the spectrum. The degree of cloudiness can be reported either as the transmittance in percent, as the optical density, or in arbitrary units. The choice of a system depends on the characteristics of the instrument used and the desires of the investigator.

CENTRIFUGATION OF JUICE SAMPLE

Although cloud is not one of the factors considered in the United States Standards for grades of frozen concentrated citrus juices, the elements of the method for its determination are included. Grade standards for frozen grapefruit concentrate (2) and for frozen blended grapefruit and orange juice concentrate (3) define the

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[2] Number in parenthesis refers to References, page 11.

conditions for the determination of the pulp content. If the supernatant liquid from this pulp determination were strained through a fine screen to remove any floating juice sacs or other materials, the resulting turbid solution could be used for cloud determination. McColloch and Rice (4) emphasize the importance of centrifuge speed on the resulting cloud values. A survey of the literature indicates that there has been no uniformity in the conditions employed for cloud determination. Many publications refer to Loeffler's method (5). In Loeffler's early work on the method for quantitative determination of cloud, centrifugation for 10 minutes at 1400 rpm was recommended, but he did not specify centrifuge size. Huggart, Moore, and Wenzel (6) used 1700 rpm for 15 minutes in an International Size 1, Type SB centrifuge, but do not specify the radius of the particular head used. DuBois and Kew (1) used 2000 rpm for 10 minutes, but do not specify the centrifuge size. Kew (7) employed 1400 rpm for 10 minutes in a centrifuge of 8-inch radius to the tip of the tube carrier. The conditions specified by Kew have been adopted as standard for cloud determinations at the Citrus Products Station. The centrifuge speeds commonly employed are in excess of those specified for pulp determination in the United States Standards previously referred to. It is suggested that 1400 rpm for 10 minutes in a centrifuge of 8-inch radius to the tip of the cups when in operating position, or its equivalent in other centrifuge sizes, be adopted as the standard conditions for cloud determinations.

Since it is essential for reproducible results that the juice be subjected to a given centrifugal force for a specific time, the following simplification of the formula for the computation of centrifugal force is suggested.

$$k = rv^2 \qquad \text{or} \qquad v = \sqrt{\frac{k}{r}}$$

In this equation k is a constant, v is the speed in hundreds of revolutions per minute, and r is the radius in inches of the centrifuge head from the center to the tip of the tube carrier when extended in a horizontal plane. For a centrifuge of 8-inch radius revolving at 1400 rpm the value of k is 1570. Table 1 gives the speeds necessary for a few common centrifuge sizes. The required speeds for centrifuges of other sizes can be computed easily with the above formulas. If k is taken as 1570, the speed computed for any given centrifuge will produce the same amount of centrifugal force which was employed by Kew. For accuracy it is essential that the centrifuge speed be measured with a good tachometer.

TABLE 1.--Centrifuge Speeds Producing Equivalent Centrifugal Force

Radius of head (inches)	*R.P.M. (hundreds)*
5.5	16.9
6.0	16.2
8.0	14.0

COLORIMETER VARIABILITY

If the specified conditions are closely adhered to, the cloud value obtained is quite reproducible on any given photoelectric colorimeter. In the absence of a reproducible standard it is possible that two different colorimeters will disagree widely on the same sample. This is true not only between instruments of different brands, but also between colorimeters of the same brand and model. The variations encountered by the authors in a series of instruments of one popular model are illustrated in Figure 1.

DESIRABILITY OF TURBIDITY STANDARD

There is one important practical reason for the significance of cloud determination to the citrus juice industry. It can be expected that optimum conditions for the storage of frozen citrus juice concentrates will not be adhered to at all times during shipment and marketing. A producer spends much time and money in preparing a superior product and builds his market on the consistently high quality of the product he ships. He is, therefore, interested in getting it into the hands of the consumer as nearly in the same condition as when it left his plant as is possible within the limitations of the transportation and marketing systems. Since the product deteriorates rapidly when it is exposed to temperatures much above 0^0 F., for example, in the ice cube compartment of a household refrigerator, and since the rate of deterioration differs from shipment to shipment, it is possible that tolerances in terms of cloud values can be established at each stage in the marketing channel which will insure retention of adequate storage life to allow it to reach the consumer in good condition. Manufacturers of certain dairy products, for example, have set limits beyond which their products cannot remain on the retailers' shelves. At least some of the producers of citrus juice concentrates are attempting to follow their products through distribution channels and to remove shipments which have become unstable before they reach the consumer. Purchasers of frozen citrus concentrates for reprocessing also are concerned with cloud values as one factor in the quality specifications of their purchases. Lack of a suitable turbidity standard has made the use of cloud values for these purposes more difficult than necessary.

Storage studies within a given laboratory also face the possibility that replacement of a critical component of the colorimeter will make it essentially a new instrument, necessitating comparison of the new values with the old. A suitable turbidity standard will facilitate this comparison.

TURBIDITY STANDARD

Various materials have been used as turbidity standards in many academic studies and for quality control purposes in a number of industries. Finely powdered glass has been proposed as such a standard, and directions for its preparation are given by Snell and Snell (8). It would be impossible to prepare such a standard in the average control laboratory, and it would be difficult to duplicate. Suspensions of barium sulfate are mentioned by Huggart, Moore, and Wenzel (6), and comparison of the turbidity of these suspensions with degree of clarification were made by visual examination. Barium sulfate standards are easily prepared and sufficiently reproducible

Variability in Response of Colorimeters of the Same Model to Suspensions of Bentonite

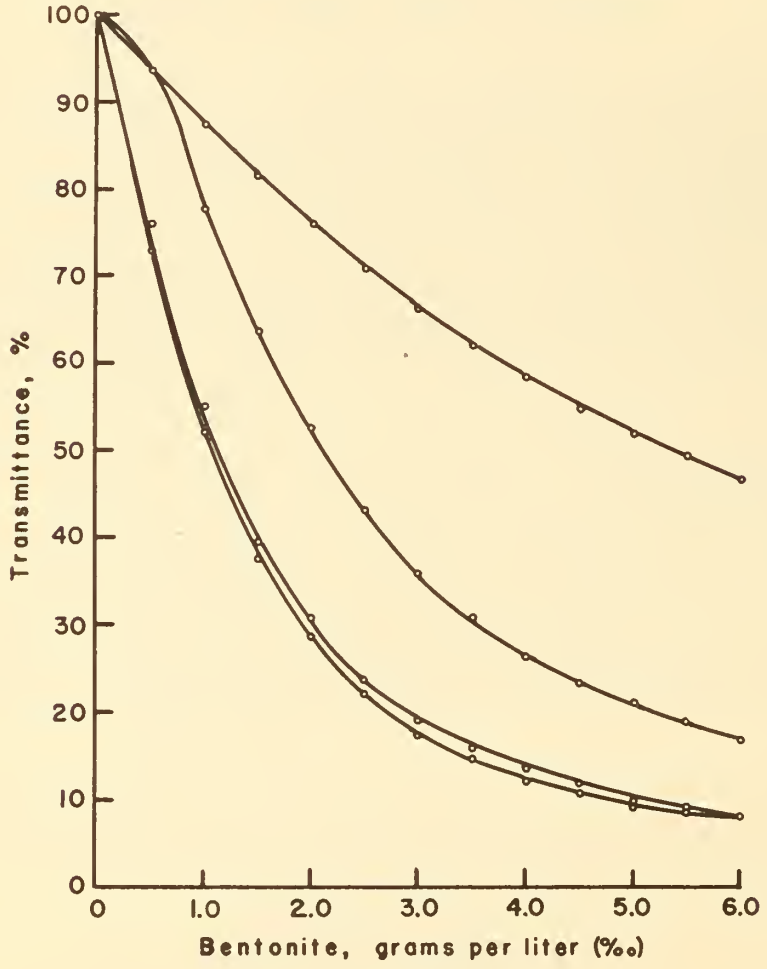

Figure 1

for visual evaluation of clarification in general terms. They are, however, neither sufficiently reproducible in turbidity for use as a primary standard, nor sufficiently stable in particle size for use as a permanent reference standard.

A number of natural clays are commercially available as finely divided powders. These are readily suspended in water. Their suspensions are stabilized by the high degree of hydration which they undergo, particularly in the presence of a trace of an appropriate electrolyte. Bentonite is a readily available clay which serves particularly well as the solid phase of a turbidity standard. When suspended in a very dilute solution of ammonium chloride the particles remain suspended for relatively long periods of time, and are redispersed readily by gently shaking after they do settle. Suspensions prepared from several batches of bentonite of the same grade obtained from different sources agreed very well in optical density when prepared from the oven dried material. These suspensions have retained their original optical densities for ten months at the time of writing.

Several grades of bentonite are commercially available. A number of these were made up to contain equal amounts of dry matter and were found to differ significantly in optical density. The finer the grade (mesh) the greater the optical density. Typical curves are presented in Figure 2.

For use as a cloud standard it is essential, therefore, that a particular grade be agreed upon. It is proposed that 325 mesh bentonite be used as such a standard.

PREPARATION OF BENTONITE STANDARD

A series of permanent standard tubes to be used for the preparation of calibration curves for the colorimeters can be made up as follows. Commercial 325 mesh bentonite is dried overnight in an oven at about 110^0 C. and cooled in a desiccator. Exactly 10.00 grams are weighed out and uniformly dispersed with the aid of a blendor or other efficient mixer in about 500 ml. of distilled water containing 0.36 g. of ammonium chloride per liter. The suspension is transferred to a 1000 ml. volumetric flask. The material remaining in the blendor bowl is rinsed into the flask with several portions of the ammonium chloride solution and finally filled to the mark with ammonium chloride solution. The bentonite suspension is transferred to a beaker equipped with a mechanical stirrer and aliquot portions of the stirred suspension are pipetted into volumetric flasks. Five ml. increments from 5 to 60 ml. and 100 ml. flasks are suitable for the preparation of a series of standards.

The aliquots are diluted to volume with the ammonium chloride solution and thoroughly mixed. These dilutions, covering the range of 0.5 to 6.0 grams of bentonite per liter in 0.5 gram increments, will be adequate for cloud determinations in single strength citrus juices.

Suitable portions of these well-mixed suspensions are transferred to matched colorimeter tubes. For permanent standards these tubes can be sealed with a glass blower's torch. They may be stored and used periodically to check the colorimeter for stability and may be used to prepare calibration curves for other instruments as required.

Turbidity of Different Bentonite Grades

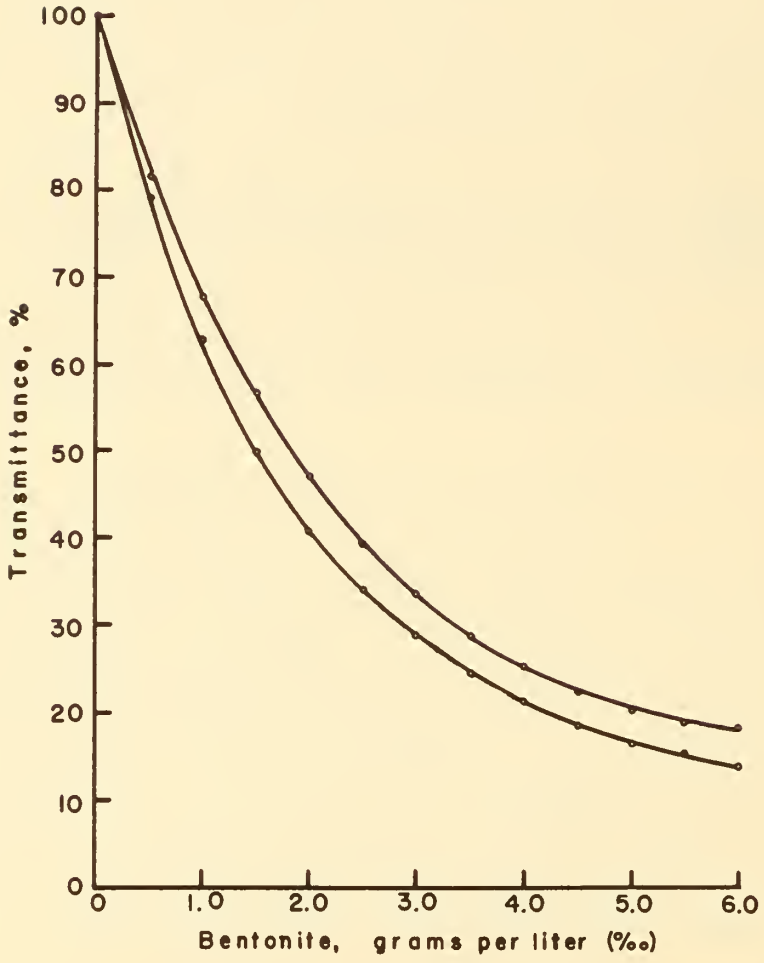

Figure 2

CALIBRATION OF PHOTOELECTRIC COLORIMETER

The percent transmittance or the optical density of these tubes is determined against a water blank with the colorimeter and filter to be used for cloud determinations. From these data the calibration curve for this particular instrument can be constructed. For convenience in routine determinations it will be simpler to prepare a table of bentonite equivalents from this calibration curve. For example, if the colorimeter dial is calibrated to read in percent transmittance, a table can be prepared giving the bentonite equivalent for each division of the dial--a total of up to 100 values. If the colorimeter is calibrated in other units, convenient small intervals can be chosen and the bentonite equivalent of each of these points read from the calibration curve.

To illustrate the operation of this system of designating cloud values, the following data were obtained on two different instruments with a series of bentonite standards sealed in matched tubes. Table 2 gives the data used in preparing the calibration curves which are presented in Figure 3.

Table 3 presents the bentonite equivalents of each of the divisions of the galvanometer dials, up to 6.00 grams of bentonite per liter, as read from the calibration curves of Figure 3.

TABLE 2.--Transmittance in per cent of suspensions of 325 mesh bentonite in water containing 0.36g. NH_4Cl per liter

Stock bentonite (ml.)	*Dil. NH_4Cl (ml.)*	*Bentonite g. per l. (o/oo)*	*Colorimeter readings*	
			Meter A	*Meter B*
0	100	0.0	100.0	100.0
5	95	0.5	71.8	64.0
10	90	1.0	58.0	48.2
15	85	1.5	46.8	37.5
20	80	2.0	38.2	28.8
25	75	2.5	31.5	22.2
30	70	3.0	26.8	17.5
35	65	3.5	23.0	14.5
40	60	4.0	20.0	12.2
45	55	4.5	17.8	10.5
50	50	5.0	15.5	9.2
55	45	5.5	14.0	8.5
60	40	6.0	12.5	8.2

Calibration Curves of Two Colorimeters for Cloud Determination in Bentonite Units

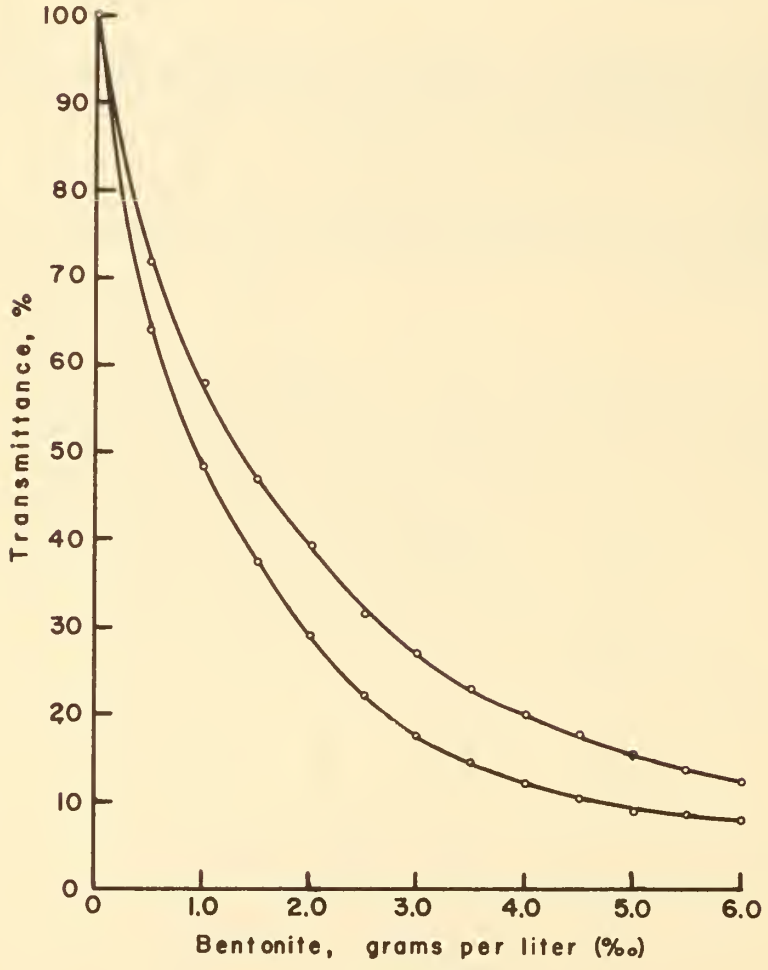

Figure 3

TABLE 3.--Bentonite equivalents of colorimeter dial divisions

BENTONITE EQUIVALENT (°/oo)			BENTONITE EQUIVALENT (°/oo)			BENTONITE EQUIVALENT (°/oo)		
% T	A	B	% T	A	B	% T	A	B
100	0.0	0.0	70	0.550	0.350	40	1.90	1.36
99	0.008	0.005	69	0.584	0.371	39	1.97	1.41
98	.016	.010	68	.618	.392	38	2.04	1.47
97	.024	.015	67	.652	.414	37	2.11	1.52
96	.032	.020	66	.686	.435	36	2.18	1.58
95	.040	.025	65	.720	.457	35	2.25	1.63
94	.048	.030	64	.760	.485	34	2.33	1.69
93	.056	.035	63	.800	.513	33	2.41	1.75
92	.064	.040	62	.840	.542	32	2.49	1.81
91	.072	.045	61	.880	.571	31	2.57	1.87
90	.080	.052	60	.920	.600	30	2.65	1.93
89	.094	.060	59	.958	.632	29	2.77	2.00
88	.108	.070	58	.996	.664	28	2.87	2.08
87	.122	.080	57	1.03	.696	27	2.98	2.15
86	.136	.090	56	1.07	.728	26	3.10	2.23
85	.150	.100	55	1.11	.760	25	3.22	2.30
84	.172	.114	54	1.16	.796	24	3.35	2.38
83	.194	.128	53	1.21	.832	23	3.50	2.47
82	.216	.142	52	1.26	.868	22	3.65	2.55
81	.238	.156	51	1.31	.904	21	3.82	2.64
80	.260	.170	50	1.36	.940	20	4.00	2.72
79	.288	.186	49	1.41	.980	19	4.17	2.84
78	.316	.202	48	1.46	1.02	18	4.37	2.95
77	.344	.218	47	1.52	1.06	17	4.62	3.07
76	.372	.234	46	1.57	1.10	16	4.85	3.22
75	.400	.250	45	1.62	1.14	15	5.14	3.40
74	.430	.270	44	1.68	1.18	14	5.45	3.59
73	.460	.290	43	1.73	1.23	13	5.86	3.80
72	.490	.310	42	1.79	1.27	12	6.0	4.03
71	.520	.330	41	1.84	1.32	11		4.32
						10		4.70
						9		5.23
						8		6.0

DESIGNATION OF CLOUD VALUE IN TERMS OF BENTONITE

These bentonite equivalent values may be referred to conveniently as "cloud values in bentonite units" and designated by the symbol "cloud, (0/00 B)." A citrus juice cloud value, then, would mean a turbidity equal to that produced by a suspension of 325 mesh bentonite at a concentration in grams per liter equal to the cloud value.

Since grams per liter equals parts per thousand, the symbol for parts per thousand, 0/00, is convenient for designating these concentrations. For example, a juice having a percent transmittance of 18 in colorimeter A (Table 3) would have a turbidity equal to that of 4.37 grams of 325 mesh bentonite per liter. In this sample, cloud would be reported as 4.37 (0/00 B).

Table 4 gives the galvanometer readings of a series of good quality as well as of partially degraded frozen orange concentrates after reconstitution. These were prepared for turbidity readings by centrifuging for 10 minutes at 1400 rpm in a centrifuge of 8-inch radius to the tip of the tube carrier. These data were obtained with the standardized photoelectric colorimeters used for the data of Table 3. In the columns adjacent to the transmittance data are given the bentonite equivalents of these juices. It can be seen that while the transmittance data differ somewhat between the two colorimeters, the bentonite equivalents are essentially identical.

TABLE 4.--Cloud values for several orange juices

SAMPLE	COLORIMETER A		COLORIMETER B	
	% T	CLOUD UNITS (°/oo B)	% T	CLOUD UNITS (°/oo B)
1	18	4.37	11	4.32
2	24	3.35	15	3.40
3	41	1.84	32	1.81
4	47	1.52	37	1.52
5	68	0.618	59	0.632
6	72	0.490	64	0.485

SUMMARY

To facilitate the comparison of cloud values obtained for citrus juices, it is suggested that the degree of turbidity of these juices be reported in terms of an arbitrary standard. Commercial bentonite, 325 mesh, is a suitable standard. It is suggested that the colorimeters intended for cloud determination be calibrated against a series of concentrations of oven dried 325 mesh bentonite dispersed in an aqueous solution containing 0.36 g. ammonium chloride per liter. The final concentrations of bentonite should cover the range 0 to 6.00 grams per liter. The citrus juice or reconstituted concentrate should be centrifuged for 10 minutes at 1400 rpm in a centrifuge of 8-inch radius (or equivalent speed in a centrifuge of different size) and screened, if necessary, to remove floating pulp particles, before determining the transmittance in a photoelectric colorimeter. The degree of cloud of the citrus juice is reported as the number of parts per thousand (grams per liter) of bentonite required to produce the same degree of turbidity. Such an artificial but reproducible standard will permit the calibration of colorimeters for cloud determination. This will markedly increase the precision of cloud comparisons by making the investigators independent of colorimeter variability.

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